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SPECIFICATION FOR TRIALLATE, TECHNICAL

UDC 632·951 TRIALLATE

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SPECIFICATION FOR TRIALLATE, TECHNICAL

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Indian Standard

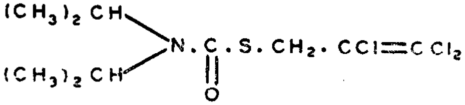
SPECIFICATION FOR TRIALLATE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 June 1980, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemicals Division Council.

0.2 Triallate, technical is used in herbicidal formulations.

0.3 Triallate is the common name accepted by the International Organization for Standardization (ISO) for S-2, 3, 3-trichloroallyl di-isopropyl thiocarbamate. The empirical and structural formulae and molecular mass are as given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{10}H_{16}Cl_3NOS$		304·7

0.4 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968, and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for triallate, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of amber coloured solid or semi-solid below 40°C, free from extraneous matter.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR TRIALLATE, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix	Cl No. of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Triallate content, percent by mass, *Min*	90	A	—
ii)	Moisture content, percent by mass, *Max*	0·3	—	4
iii)	Acidity (as H_2SO_4), percent by mass, *Max*	0·2	—	11.3.2
iv)	Material insoluble in acetone, percent by mass, *Max*	0·5	—	9

*Methods of tests for pesticides and their formulations.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS : 8190 (Part II)-1980*.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to any information as is necessary under the Insecticides Act and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;

---

*Requirements for packing of pesticides : Part II Liquid pesticides (*first revision*).

e) Triallate content, percent (*m/m*);
f) Net mass of the contents; and
g) The cautionary notice as worded in Insecticides Act and Rules.

3.2.1 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in the ' Indian Standard methods of sampling for pesticides and their formulations ' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the samples shall be drawn as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF TRIALLATE CONTENT

A-0. GENERAL

A-0.1 Either of the two methods, namely, gas chromatographic method or Parr-bomb method may be used for determination of triallate content.

---

*Specification for water for general laboratory use (*second revision*).

A-1. GAS CHROMATOGRAPHIC METHOD

A-1.1 Principle — The triallate content is determined using an internal standard on a gas chromatograph equipped with a thermal conductivity detector.

A-1.2 Apparatus

A-1.2.1 *Gas Liquid Chromatograph* — equipped with a thermal conductivity detector and a suitable recorder with an electronic integrator.

Detector	*Thermal Conductivity Detector* (*TCD*)
Column	*Glass*
	1·2 m × 4 mm packed with 10 percent carbowax 20 M and 5 percent bentone 34 on 80 to 90 mesh Anakrom ABS.
Temperature	
Column	200°C
Injection	250°C
Detector	250°C
Carrier gas	Helium
Flow rate	50 ml/min

NOTE — Operating parameter as well as attenuation and chart speed shall be adjusted to obtain optimum response and reproducibility.

A-1.2.2 *Microsyringe* — 10 µl capacity.

A-1.3 Reagents

A-1.3.1 *Standard Reference Triallate* — of known percent, 99 plus purity.

A-1.3.2 *Benzene*

A-1.3.3 *Internal Standard Solution* — *o-terphenyl or tri or tetrachloropropene.*

A-1.4 Procedure

A-1.4.1 *Preparation of the Sample Solution* — Weigh accurately sample containing equivalent to 0·2 g of triallate into a small glass stoppered flask or screw cap bottle. Add by pipette, internal standard containing 0·16 g, *o*-terphenyl solution or 0·005 g of tri or tetrachloropropene in 1·0 ml benzene. Close tightly and shake thoroughly to dissolve and extract the triallate. For coarse or granular materials shake mechanically for 30 minutes, or shake by hand intermittently for one hour,

A-1.4.2 *Preparation of Reference Standard* — Prepare reference standard solution taking 0·2 g reference standard and internal standard as given in **A-1.4.1** and make up the volume to 10 ml.

A-1.4.3 *Analysis of the Sample* — Inject 1 µl of the standard and, if necessary, adjust the instrument parameters and the volume injected to give a complete separation within a reasonable time and peak heights from half to three-fourths full scale. Proceed with the determination reinjecting each of standard and sample solution in random order.

A-1.4.4 Measure the peak heights or areas of triallate and internal standard solution.

A-1.5 Calculation

$$\text{Triallate content, percent by mass} = \frac{M_1 \times A_3 \times 100}{M_3 \times A_1} \times ACF$$

where

M_1 = mass of internal standard taken for the test,

A_3 = peak height or area corresponding to sample,

M_3 = mass of sample taken for the test,

A_1 = peak height or area corresponding to the internal standard, and

$$ACF = \frac{M_2 \times A_1 \times P_2}{M_1 \times A_2 \times P_1}$$

where

M_2 = mass of standard sample,

P_2 = percentage purity of triallate,

A_2 = peak height or area corresponding to standard sample, and

P_1 = percentage purity of the internal standard.

NOTE — Typical area correction factor with respect to *o*-terphenyl are : 1·18 for triallate.

A-2. PARR-BOMB METHOD

A-2.1 Principle — Molten sodium peroxide is an intense oxidising agent which readily converts all carbonaceous material to carbon dioxide and water. All sulphur compounds are completely oxidised to sodium sulphate in the alkaline medium of sodium peroxide. All these reactions are accomplished very quickly in a closed vessel with no contamination from external sources resulting in the transformation of all the sample to a water soluble fused mass. The thoroughness of the reaction is further promoted by the use of very finely divided potassium chlorate which serves as catalyst. After extraction sulphur is precipitated as barium sulphate and weighed.

A-2.2 Apparatus

A-2.2.1 *Peroxide Bomb Apparatus*

A-2.2.2 *Platinum Crucible* — 25-ml capacity.

A-2.2.3 *Muffle Furnace or Bunsen Burner*

A-2.3 Reagent

A-2.3.1 *Sodium Peroxide*

A-2.3.2 *Sucrose*

A-2.3.3 *Potassium Chlorate*

A-2.3.4 *Hydrochloric Acid* — Concentrated.

A-2.3.5 *Barium Chloride Solution* — 10 percent (*m/m*).

A-2.4 Procedure

A-2.4.1 To parr-bomb, add a measure full of sodium peroxide, 0·5 g of sucrose and 0·5 g of potassium chlorate. Cap it and mix the contents well by shaking.

A-2.4.2 In the extra fusion cup pour about 1/3 of sodium peroxide mixture and add about 0·2 g of sample into the centre of sodium peroxide mixture. Return 1/3 sodium peroxide mixture to the original bomb.

A-2.4.3 With a stainless steel mixing rod, mix the sample throughout the total sodium peroxide mixture. Wipe adhering sample and sodium peroxide from the rod into the reaction mixture. Add a small amount of sucrose on the top of the reaction mixture. Close the bomb and tighten the bomb case in place. Fire the bomb using electrical ignition unit. Remove the bomb from cold water bath, dry it to remove adhering water from the water bath, and remove the bomb casing. Wash the bomb with hot water into a 400-ml beaker containing 150 ml of warm water. Using tongs, place the bomb on its side in a 400-ml beaker of distilled water. Be careful that sodium peroxide dissolves and reacts at moderate rate, so it does not catch fire. Cover with a watch glass. Rinse the bomb with a stream of hot distilled water after its contents are all in solution. At moderate rate (so that it does not boil over) neutralise the beaker's contents with hydrochloric acid to methyl red with constant stirring. Filter through ordinary filter paper and wash the paper till free from acid. Concentrate the volume to about 300 ml. Heat to boiling and add 10 ml of barium chloride solution. Digest on a hot plate for about 3 hours. Half this time is sufficient if the digestion is continually just below the boiling point. Filter the hot solution through Whatman No. 42 filter paper. Wash the barium sulphate precipitate till free from chloride.

A-2.4.4 Ignite the filter paper containing the precipitate in a previously weighed platinum crucible till the filter paper turns to white ash. Heat the crucible in a muffle furnace at 800°C for about 30 minutes. Reweigh the cooled crucible.

A-2.5 Calculation

$$\text{Triallate content, percent by mass} = \frac{M_1 \times 130\ 61 \times f}{M}$$

where

M_1 = mass of barium sulphate,

$f = \dfrac{\text{Triallate in technical (GLC basis)}}{\text{Triallate in technical (sulphur basis)}}$, and

M = mass in g of the sample taken for the test.

NOTE 1 — The combination of materials used in sodium peroxide bomb has explosive properties, if wrongly handled. The operator should remain fully aware at all times of the precautions he must observe and the steps which he must take to avoid damage to the apparatus and possible injury to himself.

NOTE 2 — The operator should wear safety glasses throughout the bomb test.

NOTE 3 — Solid materials which are to be treated in a peroxide bomb should be air-dried and ground until all particles will pass 250-micron sieve. The particle size is important because the combustion reactions proceed to completion within a few seconds and burn completely within that short period.

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Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2